Performance Monitoring Protocol (QA/QC) for the Agilent 7890 High Temperature GC/FID

1 Scope

This document addresses the performance monitoring (QA/QC) of the Agilent 7890 High Temperature Gas Chromatograph with Flame Ionizations Detector (GC/FID). This document applies to personnel using the associated instrument(s)/equipment in the following discipline/category of testing: Explosives (chemistry) examinations performed at the Huntsville facility.

2 Principle

The Agilent 7890 High Temperature GC/FID is a gas chromatograph (GC) with a Multi-Mode inlet and a Flame Ionization Detector (FID). 'High Temperature' refers to the fact that the inlet is programmable to increase during a run and attain a higher temperature than an inlet is normally set, and the capillary column used is specifically designed to allow the temperature to be raised to a higher level than is typically applied to GC columns.

This performance monitoring protocol is generally based upon the manufacturer's recommendations. Definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

3 Equipment/Materials/Reagents

a. Instrumentation - Agilent 7890 Gas Chromatograph, Flame Ionization Detector, Multi-Mode inlet, and Chemstation Software (or equivalent)

b. Autosampler - Agilent ALS automated sampler, accessories, and software (or equivalent)

c. GC Column - Zebron "Inferno" ZB-1HT Capillary Column, 15 m x 0.25 mm, 0.1 µm film thickness (Phenomenex or equivalent)

d. Carrier Gas - Helium, 99.99% (high purity or equivalent)

e. Compressed air (from air purifier, compressor, tank, or equivalent)

f. Hydrogen Gas, 99.9% (or equivalent)

g. Nitrogen Gas, 99.99% (or equivalent)

h. Hexane or Cyclohexane Optima grade (or equivalent)

i. n-Paraffin Mix ~ C_{16}-C_{44} at 100 ppm (w/w) in cyclohexane (Supelco or equivalent)

j. Autosampler Syringes - 10 µL syringe (Agilent or equivalent)

k. Injection port liners - 4 mm split-splitless, tapered, with or without glass wool (Agilent or equivalent)

l. Autosampler vials - 2 mL GC vials, crimp or screw top, with or without 100-500 µL inserts (Thermo or equivalent)

m. Wash vials – 4 mL screw top without insert (Agilent or equivalent)

n. Injection port septa - standard low-bleed 11 mm (Agilent or equivalent)

4 Standards and Controls

4.1 Hi-Temp GC Testmix

The Testmix is a commercially available hexane or cyclohexane solution of n-alkanes made up of ~ C_{16}-C_{44}. This performance standard should have a concentration of approximately 10 ppm paraffin in hexane/cyclohexane and be stored at room temperature in an amber colored glass.. This solution has a shelf-life of two years. Record stock solution preparations in the Reagent Log. The Testmix is used to verify daily operating performance and continued integrity of the gas chromatograph-detector system. It will be analyzed and evaluated prior to the analysis of evidence.

5 Sampling or Sample Selection

Not applicable.

6 Procedures

6.1 Daily Checks

The following steps are to be performed daily. Enter the appropriate information in the QA/QC log.

a. Check to ensure that the GC wash vials are filled with hexane, the waste vials are empty, and all are in the appropriate positions.

b. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free disk space. Do not use if less than 100 MB remain.

c. Record the line pressure of the building helium supply (carrier gas). The regulator should read 50 psi or above. If it cannot maintain this pressure, contact the appropriate instrument support personnel. If the instrument is supplied by a gas cylinder, record the tank pressure. Change the tank if less than 100psi remain.

d. Perform an analysis of the Testmix. Open the appropriate Testmix instrument method, and verify the parameters as listed in the 'Instrumental Conditions' section of this protocol. Set up a sequence, load the autosampler with a vial containing the Testmix, and start the analysis. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the chromatogram.

e. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

6.2 As Needed Checks

The following steps are to be performed as needed. Indicate completion in the appropriate log.

a. Replace the septum within the GC injection port.

b. Replace the liner within the GC injection port.

c. Check the GC syringe in the autosampler. Replace if needed.

7 Instrumental Conditions

Refer to the "General Instrument Maintenance Protocol" for procedures on minor deviations.

Inlet/Injector

Inj Vol:	1.0 µL
Mode:	Splitless
Initial Temp:	55°C
Ramp Rate:	500°C/min
Ramp Time:	10 min
Final Temp:	400°C

Oven

Initial Temp:	55°C
Initial Time:	2.0 min
Ramp1:	30°C/min
Final Temp1:	100°C
Ramp1 Time:	0 min
Ramp2:	15°C/min
Final Temp2:	400°C
Ramp2 Time:	3.5 min
Run Time:	27.0 min
Equil Time:	0.5 min

Column

Type:	Zebron ZB-1HT (or equivalent)
Length:	15 m
Diameter:	0.25 mm
Film Thickness:	0.1 µm
Mode:	Constant Flow
Flow Rate:	1.0 mL/min
Carrier Gas:	Helium

Detector

Temperature:	420°C
Mode:	Constant makeup flow
Hydrogen flow:	40.0 mL/min
Air flow:	450.0 mL/min
Makeup flow:	30.0 mL/min
Makeup Gas:	Nitrogen

8 Decision Criteria

8.1 Testmix

Verify the results of the Testmix.

a. In order for the instrument to be considered in good operating condition, all components should generate well-resolved, Gaussian-shaped peaks with baseline separation.

b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.

c. There should be no extraneous peaks in the Testmix chromatogram greater than 5% of the tallest peak.

d. The retention times of components should not deviate by ±3% compared to previous runs of the Testmix.

9 Calculations

Not applicable.

10 Measurement Uncertainty

Not applicable.

11 Limitations

Not applicable.

12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

13 References

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (IOG 001) *Instrument Operations Group SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (IOG 002) *Instrument Operations Group SOP Manual.*

FBI Laboratory Safety Manual.

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0	10/04/18	New document which specifies instrument protocols for the Huntsville Laboratory.

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